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MEMORANDUM REPORT BRL-MR-3845

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CONDENSED-PHASE PROCESSES DURING  
SOLID PROPELLANT COMBUSTION  
I. PRELIMINARY CHEMICAL AND MICROSCOPIC  
EXAMINATION OF EXTINGUISHED PROPELLANT SAMPLES

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JUNE 1990

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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE June 1990	3. REPORT TYPE AND DATES COVERED Memorandum (Sep 88 - Sep 89)		
4. TITLE AND SUBTITLE CONDENSED-PHASE PROCESSES DURING SOLID PROPELLANT COMBUSTION. I. PRELIMINARY CHEMICAL AND MICROSCOPIC EXAMINATION OF EXTINGUISHED PROPELLANT SAMPLES		5. FUNDING NUMBERS  1 L161102AH43		
6. AUTHOR(S) Michael A. Schroder, Robert A. Fifer, Martin S. Miller, Rose A. Pesce-Rodriguez				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)		8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)  Ballistic Research Laboratory ATTN: SLCBR-DD-T Aberdeen Proving Ground, MD 21005-5066		10. SPONSORING/MONITORING AGENCY REPORT NUMBER  BRL-MR-3845		
11. SUPPLEMENTARY NOTES Published in 1989 JANNAF Combustion Meeting Proceedings				
12a. DISTRIBUTION/AVAILABILITY STATEMENT  Approved for public release; distribution unlimited		12b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) Burning propellant samples are quenched, and the burned surfaces are examined microscopically and by chemical analysis. Studies are in progress on a series of propellants including XM39, M30, JA2, RDX, HMX, HMX/PU, and HMX/polyester ("HMX2") compositions. Preliminary results on quenched samples of XM39 burned at low pressures show evidence of a liquid layer approximately 100-300 $\mu$ m thick. SEM examination reveals little if any evidence of degradation below this liquid layer. GCMS analysis shows that the stabilizer is depleted considerably in the surface layers of quenched samples, presumably by reaction with nitrogen oxides formed by decomposition of RDX and NC. HPLC results show that there is a significant increase in concentration of the mechanistically significant nitrosoamines MRDX and DRDX over the very small amounts possibly present as impurities. These results are consistent with the idea that RDX decomposition under propellant combustion conditions involves primarily (a) vaporization followed by gas-phase decomposition and/or (b) decomposition in the liquid phase to products that are almost entirely gaseous.				
14. SUBJECT TERMS Extinguishment, Quenched-Sample Analysis, Propellants, Explosives, Nitramines, Nitrate Esters, Combustion. JIS			15. NUMBER OF PAGES 28	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

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## I. INTRODUCTION

This is a progress report on work aimed at understanding the nature and importance of condensed-phase reactions in the combustion of solid nitramine and other gun propellants. Information on the nature and importance of condensed-phase reactions is needed as input for modeling studies. This information could also be very important in understanding the relationship of chemical structure and of physical properties such as melting point, phase transition temperatures, etc., to explosive and propellant behavior.

Samples have been obtained in either of two ways: (a) The propellants are burned in a low-pressure strand burner at different pressures; the sample is mounted on a massive copper block and burning is interrupted by conduction of heat away from the burning surface as the burning surface approaches the copper block, as described by Novikov and Ryzantsev;<sup>1</sup> and (b) The propellant grains are ignited with a flame in air at ambient pressure; burning is interrupted by dropping the burning grain into a beaker of water. The present report mainly emphasizes preliminary results on XM39 and its ingredients; however data are being obtained on a series of burned samples including XM39, M30, JA2, on pure HMX and RDX, on HMX-polyester ("HMX2") and on HMX/PU compositions; these data will be included in a more complete report later. In the future we plan to investigate quenching by rapid depressurization due to breaking of a rupture disk in the strand burner; the various quenching methods will then be compared.

The samples are cleaved parallel to the grain axis and the cleaved surfaces examined with a scanning electron microscope (SEM). In addition, the surface layers are removed from the extinguished propellant grains by scraping with a small, sharp knife. The resulting scrapings are analyzed by spectroscopic methods such as Fourier transform infrared spectrometry (FTIR), nuclear magnetic resonance (NMR), gas chromatography mass spectrometry (GCMS) and high performance liquid chromatography (HPLC).

The literature contains several papers describing microscopic examination of burned surfaces of propellant grains of HMX and compositions derived therefrom.<sup>2-5</sup> There are also two papers,<sup>5a,5b</sup> describing chemical analysis of the burned surface of nitrate ester propellants. However as far as we are aware chemical analysis of burned surfaces has not been applied to nitramines or nitramine propellants, although in one study<sup>5c</sup> the surface layers of a quenched RDX-polyester composition were extracted with benzene and acetone and the presence or absence of a residue under various conditions was noted; it was suggested that the variations in burning surface with particle size indicated an increase in surface temperature with decreasing particle size.

## II. EXPERIMENTAL

Propellant and ingredient samples used were standard compositions; lot numbers and grain descriptions were as follows: XM39, CI0885-200-1, Cylindrical, 1/4" X 1/4", 19-Perf.; M30, RAD-67878, Cylindrical, 1/4" X 5/8", 7-Perf.; JA2, RAD-PDI-002-1F, was received as unperforated, ca. 19"-long sticks which were cut into cylindrical, 3/8" diameter X 1/4 to 1/2 inch long grains that were used for the actual burns. The "HMX2" composition<sup>6</sup> used was a composition containing 80% HMX and 20% polyester binder. It was received as sticks 4" long and 1/4" square which were cut to lengths of approximately 1/4"

for the burns. The HMX/PU composition used was obtained from K. Resnik and R.W. Deas, and had the following composition: 80% HMX, 0.01%  $\text{TiO}_2$ , 8.075% IPDI, 2.5% TMP, and 9.415% I-35. RDX was Class A RDX and was pressed into 1/2" X 1/2" cylindrical pieces which were further cut and shaped into approximately cylindrical ca 1/4" X 1/4" pieces.

The samples were burned following one of a number of procedures; these included the following: (a) One end of the grain was ignited in air by contact with a candle, the burning end was allowed to burn for several seconds and the grain was dropped into water; (b) The grain was attached to a massive copper stub, ignited in a strand burner and allowed to burn down to the copper stub; as the burning surface approached the copper stub, quenching occurred as a result of conduction of heat away from the burning grain by the copper stub, as described by Novikov and Ryzantsev.<sup>1</sup> In addition (c), several samples of XM39 were obtained which had, for unknown reasons, extinguished spontaneously while being burned in the strand burner at a pressure of 1.0 MPa under nitrogen.

Whenever the remaining portions of the grains were substantial enough to allow it, the burned grains were cooled to dry ice temperatures and split with a splitter consisting of a knife-blade held vertical by mechanical means; when this knife was rested against the propellant grain and struck with a hammer, a clean split could be obtained (the knife was mechanically prevented from penetrating more than a small fraction of the the grain). One half of the split grain was preserved intact for microscopic examination and the surface layers of the other piece were removed by scraping with a small knife.

The acetone-soluble portions of the scrapings were analyzed by gas chromatography-mass spectrometry (GCMS) and by high performance liquid chromatography (HPLC). In some cases scraped and unscraped burned surfaces were examined by photoacoustic Fourier-transform infrared spectroscopy (FTIR). The HPLC apparatus was a Perkin-Elmer Series 4 fitted with a C-18 column and interfaced to an LC-85 spectrophotometric UV detector operating at 254 nm. Injection solvent was acetone and the eluant was 3:1 water-methanol. The GCMS apparatus consisted of a Hewlett-Packard 5970 mass selective detector (MSD) coupled to a Hewlett-Packard 5890 gas chromatograph containing an Alltech column of the following description: 30 meters long, 0.25 micron i.d., Heliflex, Bonded FSOT, RSL-150, Stock No. 13639. The carrier gas was helium. The oven program was as follows: initial hold time, 3 minutes at 50°C; Heat to 225°C at 35°C/minute; hold 15 minutes at 225°C.

Photoacoustic FTIR spectra were obtained on a Mattson Sirius 100 spectrometer using an MTEC 100 photoacoustic cell. The velocity of the interferometer moving mirror was 0.316 cm/sec. All spectra were obtained after thoroughly purging the photoacoustic cell with helium. Spectra were measured at 8  $\text{cm}^{-1}$  resolution and are the result of 32 co-added scans. Single beam spectra were ratioed to the photoacoustic spectrum of finely powdered carbon black.

The scanning electron microscope used was a JEOL 820 instrument.

### III. RESULTS

Typical HPLC curves are shown in Figures 1 and 2, and a typical GCMS curve for XM39 propellant is shown in Figure 3.

Similarly, HPLC peak areas, heights and ratios for RDX, its mononitrosoamine (MRDX) and its dinitrosoamine (DRDX) are given for burned and unburned samples of XM39 propellant and of pure RDX in Tables 1 and 2. These tables also include peak areas, heights and ratios for an unknown peak referred to as "NHMX(?)" which, based on its retention time relative to HMx (present as impurity in the RDX), could possibly be a nitrosoamine arising from HMx; however in the absence of data on an authentic sample the peak should be considered unidentified.

Tables of GCMS peak areas and heights for stabilizer (diethyl centralite) and plasticizer (ATEC) from XM39 burned-layer scrapings and of unburned XM39 are given in Tables 3 and 4; these tables also include stabilizer-plasticizer height and area ratios.

Typical photoacoustic FTIR spectra of scraped and unscraped burned surfaces, and of an unburned sample of XM39 are given in Figures 4-6.

Typical SEM Photographs of the burned surfaces of quenched and cleaved samples of XM39 and of HMx/PU are shown in Figures 7-10.

### IV. DISCUSSION

Photoacoustic FTIR Results. Figures 4-6 show photoacoustic FTIR spectra of unburned XM39 propellant (Figure 4); of the unscraped burned surface of XM39 propellant (Figure 5); and of the scraped burned surface of a sample of XM39 propellant burned in a strand burner at 1.0 MPa (Figure 6). The samples were examined by FTIR both before and after scraping, in hopes of obtaining some degree of depth profiling by examining differences between the spectra before and after scraping since the spectrum of the unscraped sample should be characteristic of the top of the burned surface, while the spectrum of the scraped sample should reflect the composition at a slight depth into the sample. All three of the spectra appear qualitatively similar, but the scraped and unscraped samples show increased intensities of the CAB peaks relative to unburned XM39; this tendency is especially noticeable in the unscraped sample. The burned samples also show a few small peaks that are not present in the unburned XM39. Attempts to identify the compounds responsible for these peaks are now in progress.

HPLC Results: Nitrosoamine Formation. Figures 1 and 2 show typical HPLC chromatograms for unburned XM39 and for the burned-layer scrapings from XM39 burned in air at atmospheric pressure. Note that Figure 1 (unburned XM39) shows only the solvent (acetone) peak at ca. 2.2 minutes, the RDX peak at ca. 6.7 minutes and a peak at ca. 3.7 minutes due to about 5-10% HMx impurity in the RDX. However, the chromatogram for the burned-layer scrapings from burned XM39 (Figure 2) shows additional peaks at around 5 and 6 minutes; these have retention times identical to peaks found<sup>7,8</sup> among the products of thermal decomposition of RDX and identified as the mononitrosamine (MRDX) and the dinitrosamine (DRDX) respectively derived from RDX. There is also a peak which occurs shortly before the HMx peak. Possibly this is due to a

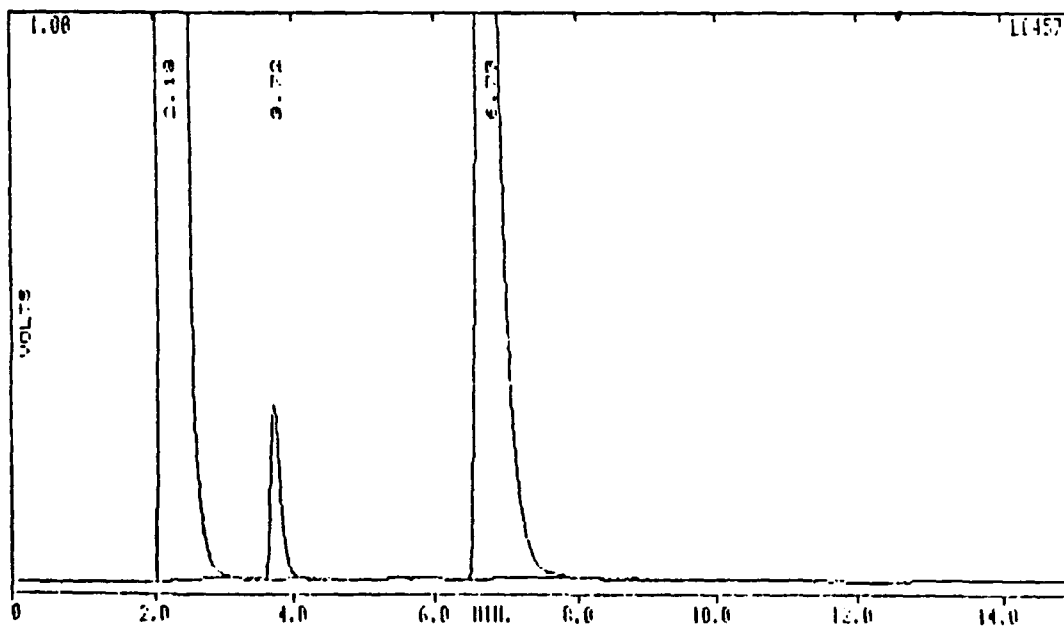


Figure 1. HPLC of Unburned XM39 Propellant in Acetone  
(Vertical, Volts; Horizontal, Minutes)

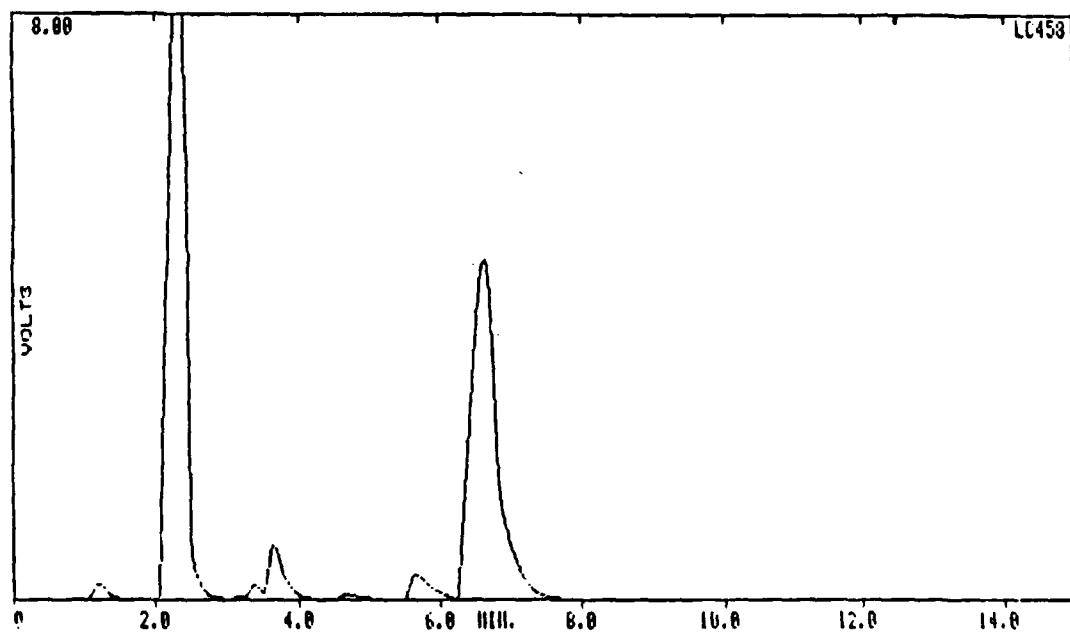


Figure 2. HPLC of Surface-Layer Scrapings of Burned XM39 Propellant  
in Acetone (Vertical, Volts; Horizontal, Minutes)

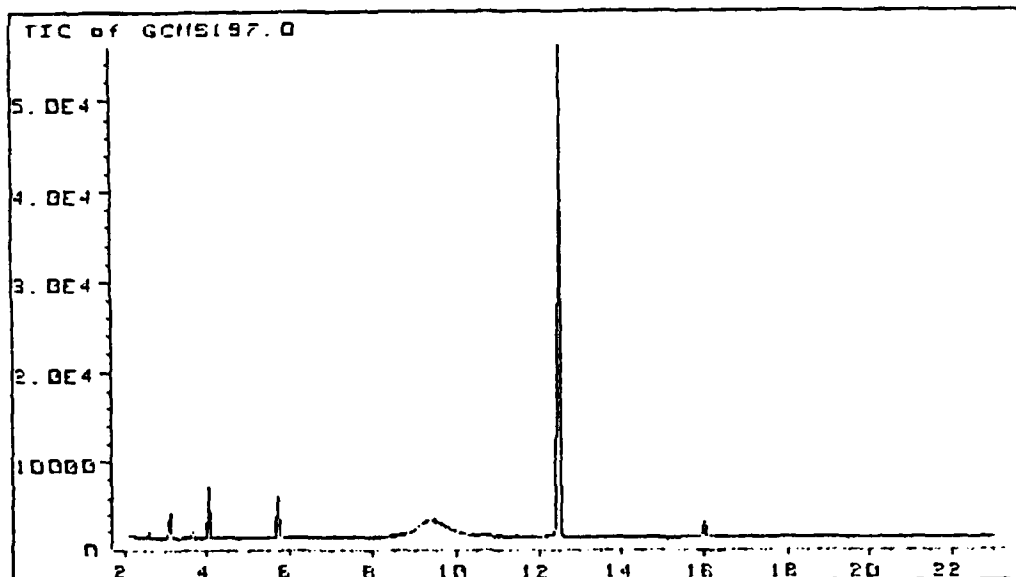


Figure 3. GCMS Chromatogram of Unburned XM39 Propellant  
(Vertical, Intensity; Horizontal, Time in Minutes)

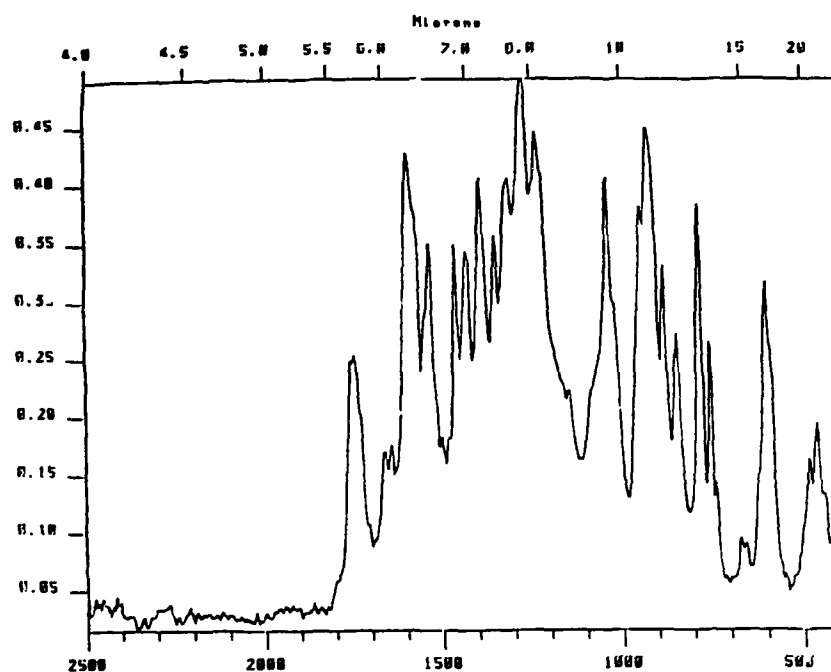


Figure 4. Photoacoustic FTIR Spectrum of Unburned XM39  
(Vertical, Intensity; Horizontal, Wavelength (Microns) or Frequency (cm⁻¹))

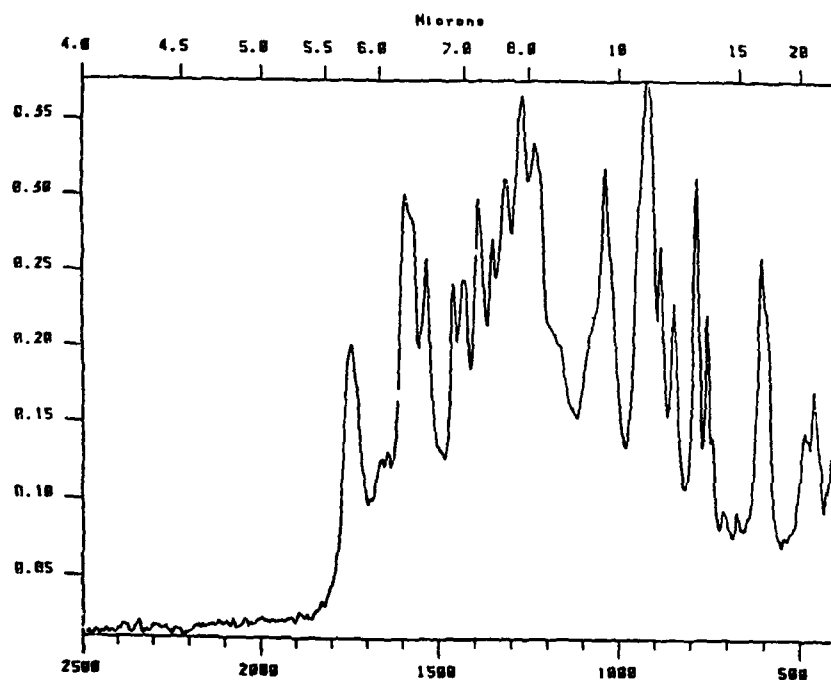


Figure 5. Photoacoustic FTIR Spectrum of Burned (1.0 MPa) XM39, Scraped Surface (Vertical, Intensity; Horizontal, Wavelength (Microns) or Frequency ( $\text{cm}^{-1}$ ))

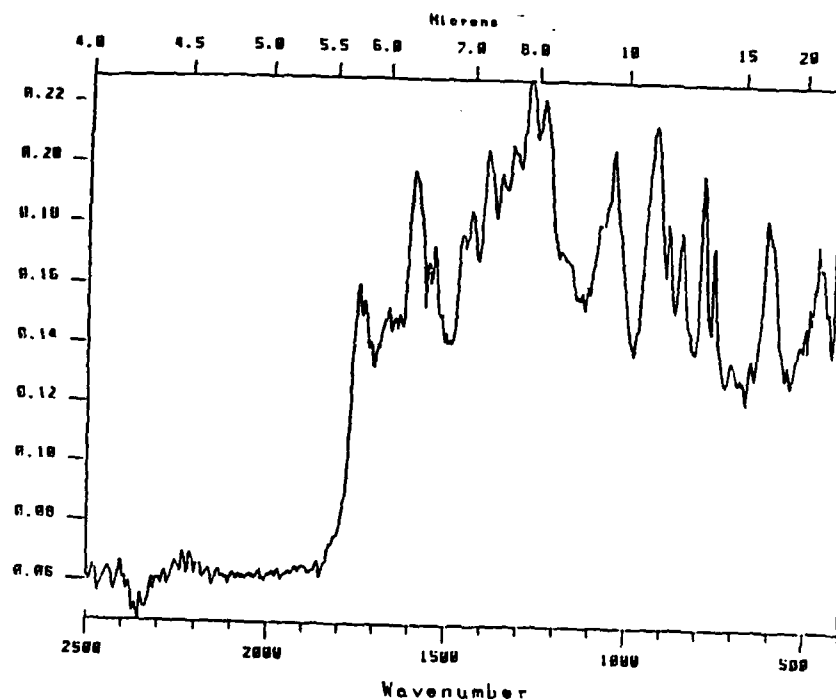


Figure 6. Photoacoustic FTIR Spectrum of Burned (1.0 MPa) Surface of XM39, Unscraped (Vertical, Intensity; Horizontal, Wavelength (Microns) or Frequency ( $\text{cm}^{-1}$ ))

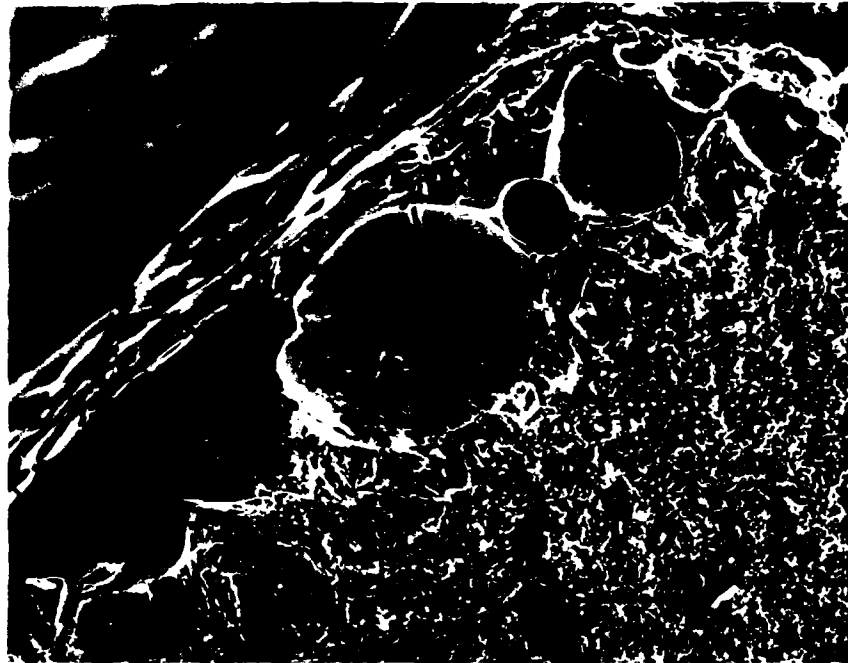


Figure 7. SEM Photograph of Quenched, Cleaved Burned Surface of XM39  
(Burned in Air, Water Quenched)



Figure 8. SEM Photograph of Quenched, Cleaved Burned Surface of XM39  
(Burned in Air, Water Quenched)

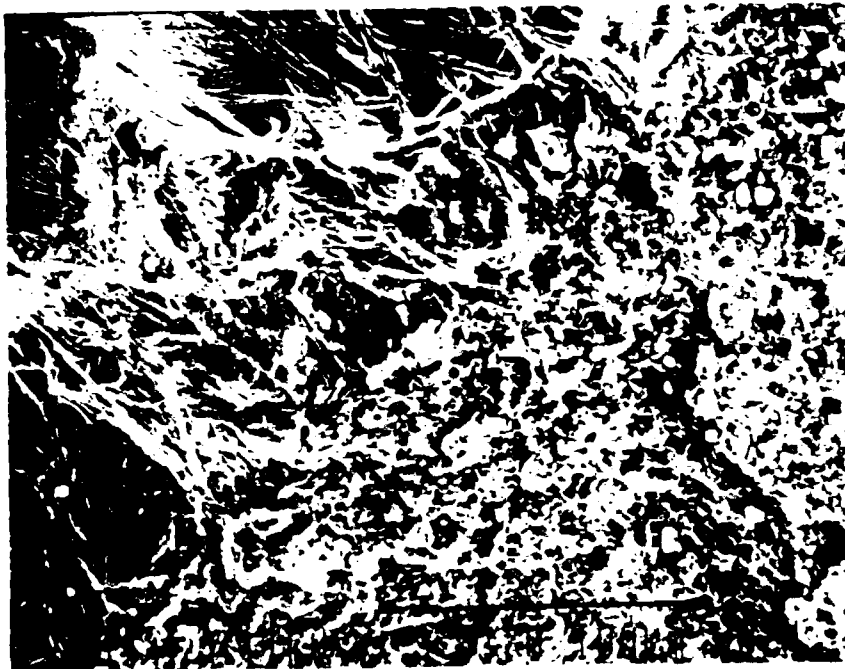


Figure 9. Close-Up of Boundary Between Solid and Liquid Layers of Quenched, Cleaved Burned Surface of XM39 (Burned in Air, Water Quenched)



Figure 10. SEM Photograph of Quenched, Cleaved Burned Surface of HMX/PU (Burned in Air, Water Quenched)



Table 1. Table of Chromatographic Peak Areas and Area Ratios for RDX, HMX, and Nitrosoamine Peaks.

Burn No.	Run (LC--)	Sample	Areas					Area Ratios x 100			
			NHMX(?)	HMX	ORDX	MRDX	RDX	NHMX?/HMX	ORDX/RDX	MRDX/RDX	HMX/RDX
081589A	452	XM39 (Air, lat., WO) BL <sup>a</sup>	8412	48409	3689	24108	645244	17.3	0.6	3.7	7.5
081589H	457	XM39 (Unburned) <sup>b</sup>	0	32577	0	0	465275	0	0	0	7.0
081589G	458	XM39 (Air, lat., WO) BL <sup>a</sup>	23267	107645	13694	72417	1246698	21.4	1.1	5.8	8.6
090889E	463	RDX (Air, lat., WO) BL <sup>a</sup>	1621	103991	0	1171	2559890	1.6	0	0.05	4.1
090889E	464	RDX (Air, lat., WO) BL <sup>a</sup>	510	35804	0	1122	379198	1.4	0	0.3	9.3
090889K	465	RDX (Air, lat., WO) BL <sup>a</sup>	0	26087	0	864	248194	0	0	0.3	10.5
090889Q	466	RDX (Unburned) <sup>b</sup>	0	36853	0	0	698763	0	0	0	5.3
090889E/K	470	RDX (Air, lat., WO) FM <sup>a, c</sup>	12475	292005	3682	69839	2306358	4.3	0.2	3.0	12.7
090889E/K	471	RDX (Air, lat., WO) FM <sup>a, c</sup>	6706	170566	3722	42819	626662	3.9	0.6	6.8	27.2
081589A	473	XM39 (Air, lat., WO) BL <sup>a</sup>	2004	14552	503	3815	107531	13.8	0.5	3.5	13.5
89090603	477	XM39 (SB, 1.MPa, SE) BL <sup>d</sup>	0	7211	1661	14019	849473	0	0.2	1.7	0.8
89090701	478	XM39 (SB, 1.MPa, SE) BL <sup>d</sup>	0	13951	1290	9023	144415	0	0.9	6.2	9.7
89090702	479	XM39 (SB, 1.MPa, SE) BL <sup>d</sup>	980	30515	2188	6399	284099	3.2	0.8	2.3	10.7
N/A	480	XM39 (Unburned) <sup>b</sup>	0	25821	0	0	426220	0	0	0	6.1

- a. Burned-layer scrapings from a grain burned in open air at atmospheric pressure and quenched in water.  
b. Unburned material.  
c. Material thrown off by an RDX grain burning in open air at atmospheric pressure.  
d. Burned-layer scrapings from a grain that self-extinguished while burning in a strand burner at 1.0 MPa.

Table 2. Table of Chromatographic Peak Heights and Height Ratios for RDX, HMX, and Nitrosoamine Peaks.

Burn No.	Run (LC--)	Sample	Heights					Height Ratios x 100			
			NHMX(?)	HMX	DRDX	MRDX	RDX	NHMX?/HMX	DRDX/RDX	MRDX/RDX	HMX/RDX
081589A	452	XM39 (Air, lat., WO) BL <sup>a</sup>	813	4149	284	1363	30147	19.6	0.9	4.5	13.8
081589H	457	XM39 (Unburned) <sup>b</sup>	0	3115	0	0	25612	0	0	0	12.2
081589G	458	XM39 (Air, lat., WO) BL <sup>a</sup>	2002	7495	709	3414	47110	26.7	1.5	7.2	15.9
090889E	463	RDX (Air, lat., WO) BL <sup>a</sup>	150	4744	0	103	68334	3.2	0	0.2	6.9
090889E	464	RDX (Air, lat., WO) BL <sup>a</sup>	60	1629	0	79	7326	3.7	0	1.1	22.2
090889K	465	RDX (Air, lat., WO) BL <sup>a</sup>	0	1253	0	77	5670	0	0	1.4	22.1
090889Q	466	RDX (Unburned) <sup>b</sup>	0	1858	0	0	17685	0	0	0	10.5
090889E/K	470	RDX (Air, lat., WO) FM <sup>a, c</sup>	1187	16631	263	2910	84682	7.1	0.3	3.4	19.6
090889E/K	471	RDX (Air, lat., WO) FM <sup>a, c</sup>	741	9625	226	1851	20231	7.7	1.1	9.1	47.6
081589A	473	XM39 (Air, lat., WO) BL <sup>a</sup>	347	1670	85	459	9346	20.8	0.9	4.9	17.9
89090603	477	XM39 (SB, 1.MPa, SE) BL <sup>d</sup>	0	842	162	600	17803	0	0.9	3.4	4.7
89090701	478	XM39 (SB, 1.MPa, SE) BL <sup>d</sup>	0	822	137	539	4705	0	2.9	11.5	17.5
89090702	479	XM39 (SB, 1.MPa, SE) BL <sup>d</sup>	113	1649	156	462	8531	6.9	1.8	5.4	19.3
N/A	480	XM39 (Unburned) <sup>b</sup>	0	1193	0	0	13373	0	0	0	8.9

- a. Burned-layer scrapings from a grain burned in open air at atmospheric pressure and quenched in water.  
b. Unburned material.  
c. Material thrown off by an RDX grain burning in open air at atmospheric pressure.  
d. Burned-layer scrapings from a grain that self-extinguished while burning in a strand burner at 1.0 MPa.

Table 3. Table of Stabilizer and Plasticizer Peak Areas and Ratios  
from Burned Surface and from Unburned XM39

Burn No.	Run No. (GCMS---)	Sample	Areas		Ratio (X100)
			Plast.	Stab.	
N/A	197	XM39 (Unburned) <sup>a</sup>	2191335	85785	3.9
N/A	200	XM39 (Unburned) <sup>a</sup>	651625	25480	3.9
050889A	210	XM39 (Burned Surf.) <sup>b</sup>	769750	0	0
050889A	211	XM39 (Burned Surf.) <sup>b</sup>	1856745	0	0
050889C	212	XM39 (Burned Surf.) <sup>b</sup>	1328024	0	0
081589A	216	XM39 (Burned Surf.) <sup>b</sup>	1466484	24162	1.6
081589G	220	XM39 (Burned Surf.) <sup>b</sup>	2249097	8809	0.4
081589M/N	224	XM39 (Unburned) <sup>a</sup>	899588	38870	4.3
89090603	232	XM39 (Self-Ext.1 .MP) <sup>c</sup>	1246449	29261	2.3
89090701	234	XM39 (Self-Ext.1 .MP) <sup>c</sup>	464287	0	0
89090702	235	XM39 (Self-Ext.1 .MP) <sup>c</sup>	1175263	23431	2.0
89090701	236	XM39 (Self-Ext.1 .MP) <sup>c</sup>	1458581	13053	0.9
N/A	237	XM39 (Unburned) <sup>a</sup>	1210499	55854	4.6
N/A	238	XM39 (Unburned) <sup>a</sup>	2059454	93859	4.6
N/A	239	XM39 (Unburned) <sup>a</sup>	659455	21148	3.2

a. Unburned material.

b. Burned-layer scrapings from a grain burned in air at atmospheric pressure and quenched in water.

c. Burned-layer scrapings from a grain that self-extinguished while burning in a strand burner at 1.0 MPa.

Table 4. Table of Stabilizer and Plasticizer Peak Heights and Ratios  
from Burned Surface and from Unburned XM39

Burn No.	Run No. (GCMS---)	Sample	Heights		Ratio (X100)
			Plast.	Stab.	
N/A	197	XM39 (Unburned) <sup>a</sup>	53000	2000	3.8
N/A	200	XM39 (Unburned) <sup>a</sup>	21000	500	2.4
050889A	210	XM39 (Burned Surf.) <sup>b</sup>	17000	0	0
050889A	211	XM39 (Burned Surf.) <sup>b</sup>	31000	0	0
050889C	212	XM39 (Burned Surf.) <sup>b</sup>	29000	0	0
081589A	216	XM39 (Burned Surf.) <sup>b</sup>	30000	600	2.0
081589G	220	XM39 (Burned Surf.) <sup>b</sup>	40000	200	0.5
081589M/N	224	XM39 (Unburned) <sup>a</sup>	23000	900	3.9
89090603	232	XM39 (Self-Ext.1.MP) <sup>c</sup>	25000	600	2.4
89090701	234	XM39 (Self-Ext.1.MP) <sup>c</sup>	14000	0	0
89090702	235	XM39 (Self-Ext.1.MP) <sup>c</sup>	30000	500	1.7
89090701	236	XM39 (Self-Ext.1.MP) <sup>c</sup>	40000	350	0.9
N/A	237	XM39 (Unburned) <sup>a</sup>	34000	1000	2.9
N/A	238	XM39 (Unburned) <sup>a</sup>	44000	1970	4.5
N/A	239	XM39 (Unburned) <sup>a</sup>	22000	400	1.8

a. Unburned material.

b. Burned-layer scrapings from a grain burned in air at atmospheric pressure and quenched in water.

c. Burned-layer scrapings from a grain that self-extinguished while burning in a strand burner at 1.0 MPa.

nitrosoamine derived from HMX; it is referred to in Tables 1 and 2 as "NHMX(?)". However, in the absence of an authentic sample, this peak should be considered unidentified.

Tables 1 and 2 show the relative intensities of the HMX, RDX and nitrosoamine HPLC peaks, estimated respectively from peak areas and peak heights. Since the response factors<sup>7,9</sup> for these compounds are similar, these should provide a rough, order-of-magnitude estimate of the amounts of nitrosoamines formed, relative to RDX and HMX. It is thus estimated that the nitrosamines are present in amounts as high as 1-10% of the unreacted RDX in some cases.

The formation of nitrosoamines in amounts as large as these near the the burning surface of nitramine propellants and compositions seems quite significant with regard to chemical mechanisms; this will be discussed below under "Conclusions". In addition to the mechanisms discussed there, another possibility that should be considered arises from the occurrence,<sup>16</sup> in at least some samples of RDX, of GC peaks with the same retention times as MRDX and TRDX, consistent with the presence of these materials as trace impurities. Since MRDX decomposes about 10% slower than RDX at 180°C in benzene under pressure,<sup>8</sup> it is difficult to rigorously rule out the possibility that trace amounts initially present could accumulate to larger concentrations in the burning surface. Even though we did not detect any nitrosoamines in the lots of XM39 used in the present work, the possibility of the presence of amounts too small to detect by our methods should be kept in mind.

GCMS Results: Stabilizer Depletion. Figure 3 shows a typical GCMS chromatogram for XM39 propellant. The peaks at less than six minutes arise from impurities in the injection solvent (acetone). The main features are: (a) a large, broad peak at around 8-9 minutes which is believed to contain primarily gaseous decomposition products of RDX and NC; (b) a very sharp, intense peak at ca. 12.5 minutes which was identified by its mass spectrum and retention time as being due to the plasticizer ATEC; and a very weak but still sharp peak at ca. 15.9-16.0 minutes which was identified by its mass spectrum and retention time as being due to the stabilizer diethyl centralite.

Tables 3 and 4 show the intensities of these peaks, relative to each other, as measured by the peak areas and peak heights respectively, for a number of burned and unburned samples. Note that the intensity of the stabilizer peak, relative to the plasticizer, is considerably less for the burned-layer chromatograms than for the chromatograms from unburned XM39. This is seen both from relative areas (Table 3) and relative heights (Table 4). This suggests that the amount of stabilizer present in the surface/liquid layers is less than in the unburned propellant; this would be consistent with its removal in the liquid layer by reactions with nitrogen oxides formed by decomposition of RDX and NC. Possibly this removal occurs by mechanisms similar to those involved in stabilization of the propellant by removal of trace amounts of nitrogen oxides and acids.

It has been reported,<sup>5a</sup> in a paper describing HPLC analysis of burned layer samples from nitrate ester propellants, that if the propellant specimens were not sampled soon after quenching, the nitroglycerine (NG) tended to diffuse into the NG-depleted zones near the surface from the deeper layers.

It seems unlikely that such an effect is entirely responsible for the apparent stabilizer depletion seen in Tables 3 and 4. This follows from the fact that the burned samples corresponding to GCMS's 210, 211, and 212 (lines 3-5 of both tables) which all showed no detectable stabilizer remaining were scraped on the same day that burning was carried out. The remaining burned samples, which were scraped one week or more after burning, did show some stabilizer, albeit in most cases considerably less than in the case of unburned XM39.

It should be remembered however that what is actually being measured is not the absolute amount of stabilizer but the ratio of stabilizer to plasticizer; in principal the observed effect could be produced as well by an increase in plasticizer as by a decrease in stabilizer. However it is difficult to see why the ATEC (plasticizer) should migrate to the burning surface appreciably faster than the diethyl centralite (stabilizer), or vaporize from it more slowly; both of these possibilities will be investigated further.

SEM Results: Structure of Liquid Layer. Typical SEM Photographs of the burning surfaces of burned, quenched and cleaved grains of XM39 are shown in Figures 7-9. These samples were burned at atmospheric pressure in air, then quenched by dropping them into water.

Figures 7 and 8 are two different views of the burned surface from XM39 under the above conditions. These samples show considerable evidence for the presence during combustion of a liquid layer, about 100-300 microns thick; this layer solidified with recrystallization of RDX after quenching with water. Evidence of the liquid layer includes numerous bubbles and the formation of what appear to be crystals, especially in the area immediately adjacent to the unburned propellant, suggesting that crystallization may have been seeded by the RDX crystals in the unburned propellant. The liquid layer seems to be overlain in places by another layer, possibly of molten binder.

Note also that there appears to be no evidence for any change in the structure of the unburned propellant before melting, as the structure below the liquid layer appears to remain constant right up to bottom of the liquid layer. This follows especially from Figure 9, which is an enlarged view of the boundary between the two layers.

Figure 10 is an SEM photograph of the quenched, cleaved (air, ambient pressure, water quenched) burned surface of a grain of HMX/PU composition. The liquid layer is thinner than in the case of the RDX composition XM39. Some small bubbles can be seen. Some sites appear to show crystallization of oxidizer, but this is much less obvious than in the case of XM39.

## V. CONCLUSIONS

The above allows the construction of at least a partial picture of the combustion process, at least in the case of XM39 in air at atmospheric pressure.

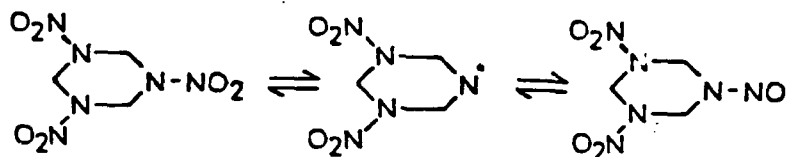
First, judging from the apparent lack of change in the solid material below the liquid, there is little solid-state reaction. Most of the decomposition takes place after the oxidizer (RDX) melts. Extensive liquid-state decomposition to gaseous products is suggested by the bubbles observed

in the SEM photos of the liquid layer, although it is difficult to be sure how much oxidizer escapes to the vapor phase before decomposing. This is consistent with the observed tendency<sup>13,17</sup> for RDX to decompose faster in the liquid than in the solid state.

Our detection, in the present work, of increased concentrations of nitrosoamines in the surface layers is also consistent with decomposition in the liquid layer, in view of the reports<sup>18,19</sup> of mass spectrometric detection of a peak at  $m/e$  132, characteristic of the nitrosoamine fragment  $\cdot\text{CH}_2\text{N}(\text{NO})\text{CH}_2\text{N}(\text{NO}_2)$ ; when the temperature of decomposing RDX rises above its melting point.

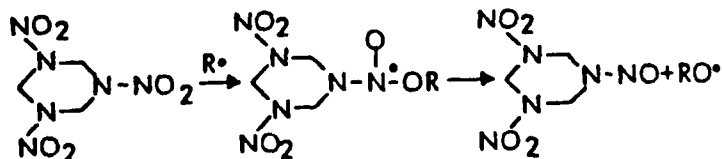
The chemical mechanisms involved in the liquid-state decomposition remain uncertain, although it is possible to make some comments about them based on the results of the present work. First, the apparent stabilizer-depletion effect reported above is consistent with the idea that the liquid-phase combustion chemistry must in some way involve formation of nitrogen oxides such as NO and  $\text{NO}_2$ .

Second, the observation, in the present work, of nitrosoamines in significant amounts in the the burning surface of nitramine propellants seems quite significant with regard to chemical mechanisms, since it is easiest to explain by two mechanisms.<sup>10-15</sup> These include: (see especially Reference 10) (a) initial  $\text{N}-\text{NO}_2$  cleavage followed by recombination of the resulting nitrogen-centered radical with NO (Scheme I); and



SCHEME I

(b) Oxygen abstraction from RDX or HMX, possibly by a free radical formed in the decomposition (Scheme II).



SCHEME II

It is impossible to distinguish between these mechanisms without further information; some possible means of distinguishing between them are discussed below. Behrens<sup>20</sup> has recently described isotope scrambling studies on thermal decomposition of mixtures of unlabeled and fully-<sup>15</sup>N-labeled HMX; the only product discussed was the mononitrosoamine, which was unscrambled. This suggests that this compound is formed without breaking of the N-N bond, most likely by free-radical oxygen abstraction (Scheme II). Note however that this result pertains to thermal decomposition, not to actual combustion. It would be extremely interesting to repeat the experiments described in the present report on a mixture of unlabeled and fully-labeled RDX, and examine the resulting nitrosoamines for scrambling during the combustion process.

## VI. WORK NEEDED/FUTURE PLANS

After completing the chemical analysis and microscopic examination of the burned samples now on hand, we plan to carry out runs in which quenching is carried out by depressurization, and compare the results obtained by the various quenching methods. Also, optical microscopic examination of the burned surfaces will be carried out, particularly in view of the possibility that studies of color changes and variations may yield information on the occurrence (or lack thereof) of chemical changes in the solid below the liquid layer. Ways of obtaining improved depth profiling of the burned layers will also be explored; these include microabrasive blasting, microtoming, solvent-dipping, and improved scraping procedures. Other propellant formulations will also be examined.

Isotope-scrambling studies are needed. These would involve use of mixtures of unlabeled RDX or HMX with RDX or HMX labeled with nitrogen-15 in all nitrogens, both in the ring and in the nitro groups. Use of these mixtures would lead to scrambled nitrosoamines (and also to scrambling in the starting RDX and/or HMX) if the recombination mechanism (Scheme I) were operating, but to unscrambled nitrosoamines if the oxygen-abstraction mechanism (Scheme II) were operating. Partial scrambling would mean that both mechanisms were operating to some degree. Note however that while formation of fully-scrambled nitrosoamines would provide no evidence for oxygen abstraction, it would not necessarily rule it out, since the scrambling could have taken place by further N-N cleavage equilibria before or after formation of the nitrosoamines.

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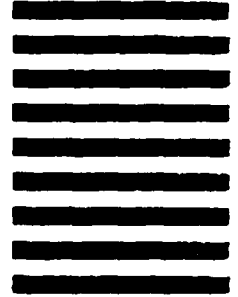
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